

# Color enhancement of refined-bleached used vegetable oils as dielectric liquid: two-level factorial design approach

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## ABSTRACT

Number of findings have shown that the used vegetable oils (UVOs) properties can be enhanced by changing their chemical structure and can be utilized as dielectric liquid in oil-immersed transformers. However, earlier researchers used the one-factor-at-a-time (OFAT) method for their experimental design approach. Nevertheless, they failed to consider the possibility that combining the mixing process parameters at the highest ratios could produce a more favorable outcome. Hence, in this study, two-level ( $2^k$ ) factorial design is applied to achieve the highest color reduction of UVOs through chemical refining process known as refined-bleached UVOs (RBUVOs). The involved process parameters are oil temperature, mixing speed and mixing time. Based on the results of  $2^3$  factorial design, it is found that mixing time and oil temperature has the most significant effects on color reduction, with a percentage contribution of 35.00% and 32.51%, respectively. The result also shows that the best mixing process parameters of RBUVOs were oil temperature (80 °C), mixing speed (1,000 rpm) and mixing time (60 min). These resulted in the highest color reduction of RBUVOs by 79.27%.

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## 1. INTRODUCTION

Natural ester insulating (NEI) oils come in two varieties: virgin vegetable oils (VVOs) and used vegetable oils (UVOs). UVOs have essentially used cooking oils that can be acquired after the oils have been used for frying meals. Like VVOs, UVOs are a renewable resource and have excellent potential as transformer-insulating oils. In oil-immersed power transformers, UVOs have demonstrated considerable potential as fuels, biolubricants, and coolants. Note that UVOs do have certain drawbacks. These include low pour points [1], low oxidation stability [2], low impulse breakdown strength [3]–[5], high viscosity [6], greater acidity [7], and color appearances that are excessively dark and above the permissible color value (color number) of 1.0 [8]. Some energy utilities have chosen not to utilize NEI oil in oil-immersed power transformers as a result of these drawbacks. One of the NEI oils' physical properties that need to be periodically assessed to comprehend the degradation of the insulating liquid is color. The highest allowable limit of color is 1.0 and light green, respectively, according to the IEEE guide for acceptance and maintenance of natural ester fluids in transformers (IEEE C57.147:2008) and American standard testing and

material (ASTM) D1500 standard [8], [9]. Color is a measurement of the transformer insulating oil's level of pollution. Generally speaking, a change in color indicates that the insulating oil has degraded and been polluted [10]. In order to remove impurities (such as color pigments, gum traces, soap traces, metals, natural antioxidants, oxidation products, and acidic components) from NEI oils, several researchers have modified the chemical structure of VVOs by combining techniques like multi-stage filtration with adsorbents, refining and reconditioning, as well as esterification [8], [11]–[13].

The majority of research on UVOs to date has concentrated on their potential for use in biodiesel production and as a biolubricant [14]–[16]. The effects of chemical treatment (neutralization and bleaching) and refinement (neutralization and bleaching with synthetic silicate adsorbent process) on UVOs as a potential dielectric fluid for power transformers have only been studied in one published report to date. Still, the mixing process parameters (specifically the oil temperature, mixing speed, and mixing time) have not been optimized specifically for UVOs [8]. Additionally, the high temperature used during refining may cause beneficial bioactive chemicals to degrade easily [17]. Additionally, to find the ideal level of mixing process parameters that will enhance the oil characteristic, previous research solely used the one-factor-at-a-time (OFAT) method. The OFAT method, however, requires plenty of test runs or experiments to estimate the effect that might produce better results. Since it takes a lot of time and money to complete all the experiments, the requirement for a large number of test runs is generally undesirable [18], [19]. Therefore, the mixing process parameters are required to achieve the maximum effect for various insulating oils in order to produce the greatest amount of UVOs with the highest quality attributes while simultaneously removing the undesired component from the crude oil.

In this study, the parameters of the mixing process, including i) the speed of stirring used to mix the UVOs with the adsorbent (fuller's earth and synthetic silicate), ii) the temperature of the UVOs when they are mixed with the adsorbent (fuller's earth and synthetic silicate), and iii) the mixing time when they are mixed with the adsorbent (fuller's earth and synthetic silicate), should be refined using a two-level factorial design of (RBUVOs). Analysis of variance (ANOVA) is utilized to confirm the outcomes of this technique. The model's effectiveness is then tested using ANOVA to determine whether it is adequate for predicting the color reduction of the RBUVOs as a function of the mixing process factors (particularly oil temperature, mixing speed, and mixing duration).

## 2. METHOD

### 2.1. Sample preparation

For the neutralization, bleaching, and treatment processes in this investigation, UVOs obtained from hotels, caterers, and restaurants were chosen. The 1 L UVO was first pre-processed by heating it for 10 min at 110 °C to roughly 400 ppm water content. Once the temperature dropped to 60 °C, heated UVOs were kept at ambient temperature. UVO was then neutralized in a 2 L beaker with sodium hydroxide (NaOH) solution (normality 3.0N). Using a hot plate magnetic stirrer, the mixture was stirred at 500 rpm for 5 min at a temperature of 70 °C. After that, the combined samples were covered in aluminum foil and kept in a dry, cool place for 24 h. The combined samples were divided after 24 h into two layers: neutralized UVO (NUVO) (top layer) and soap stock (bottom layer). Seven process of washings thoroughly cleaned the neutralized oil to remove any traces of soap stock and NaOH. Before bleaching and treatment, the mixed samples were filtered to remove the soap stock and transferred the NUVO to another 2 L beaker. The samples were made using fuller's earth and synthetic silicate adsorbent and were based on the two-level (2n) factorial design matrix generated from the screening process. In order to greatly increase the color reduction of the employed vegetable oils, the volume concentrations of both adsorbents were maintained at 20 wt.% and 15 wt.%, respectively. New refined-bleached UVOs (RBUVOs) and mixing process parameters were carried out during the mixing process at various settings, as shown in Table 1. To eliminate the sludge (i.e., a substance composed of fuller's earth and synthetic silicate adsorbent and polar components present in the RBUVOs) after the mixing process, the RBUVOs with mixing procedure parameters mixes was separated using whatman filter paper (pore size: 0.2 micron). The oil sample was put into a glass bottle once the filtration procedure was finished, and it was left there for 24 h before the color reduction test.

### 2.2. Determination of color reduction percentage

Using a UV-Vis spectrophotometer (UVmini-1240, Shimadzu Corporation, Japan) and heptane as a standard. The color reduction was evaluated in accordance with ASTM D6802-02 standard test technique to determine the absorbance values of the oil at a wavelength range from 360 to 600 nm [20], [21]. The formula used to determine the percentage of color reduction [17], [22], [23] is as (1).

$$\text{Color reduction (\%)} = \left( \frac{A_0 - A_1}{A_0} \right) \times 100 \quad (1)$$

where  $A_0$  is the absorbance of NUVO, and  $A_1$  is the absorbance of RBUVOs with a maximum level of mixing process parameters.

### 2.3. Design of experiments

Version 10.0.8.0 of design-expert software was utilized for the experiment design (Stat-Ease, Inc., Minneapolis, USA). The effects of each mixing process parameter, including oil temperature, mixing speed, and mixing time, were evaluated on the NUVO using the  $2^k$  factorial design. Low, medium, and high levels are denoted by -1, 0, and +1, respectively, in Table 1, which also displays the level of the oil temperature, mixing speed, and mixing time variables. There are thirteen test runs along with a 23-factorial design matrix and five center points that were utilized to filter the factors. According to the 23-factorial design matrix, the color reduction test was conducted. The color reduction of RBUVOs was studied using analysis of variance (ANOVA) to determine the impacts of the oil temperature, stirring rate, and mixing time (factors 1, 2, and 3, respectively). In order to maximize the color reduction of the RBUVOs, the response surface plot was then used to optimize the mixing process parameters (i.e., oil temperature, mixing speed, and mixing time).

Table 1. Two-level factorial design matrix for three independent variables obtained from design-expert

Experimental trial no.	Variable code		
	A: Oil temperature (°C)	B: Mixing speed (rpm)	C: Mixing time (min)
1	80 (+1)	1,000 (+1)	30 (-1)
2	70 (0)	750 (0)	45 (0)
3	70 (0)	750 (0)	45 (0)
4	70 (0)	750 (0)	45 (0)
5	60 (-1)	500 (-1)	30 (-1)
6	60 (-1)	500 (-1)	60 (+1)
7	60 (-1)	1,000 (+1)	60 (+1)
8	70 (0)	750 (0)	45 (0)
9	80 (+)	500 (-1)	60 (+1)
10	60 (0)	1,000 (+)	30 (-1)
11	70 (0)	750 (0)	45 (0)
12	80 (+1)	500 (-1)	30 (-1)
13	80 (+1)	1,000 (+1)	60 (+1)

### 2.4. Screening process

In this phase, all the test run data is analyzed, and the optimum points are estimated. A regression model was created based on the screening process findings to forecast color reduction as a function of oil temperature, stirring rate, and mixing time. Here, the oil temperature, the rate of stirring, and the amount of mixing time are the independent factors, while color reduction is the response variable. To verify the regression model's statistical significance, ANOVA was employed. The following is the regression (2):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 \quad (2)$$

in which  $y$  denotes the response variable (predicted variable),  $x_1$  and  $x_2$  resembles the independent variables (factors),  $x_1 x_2$  denotes the interaction between factors  $x_1$  and  $x_2$ ,  $\beta_1$  and  $\beta_2$  denotes the coefficients associated with factors  $x_1$  and  $x_2$ , respectively,  $\beta_{12}$  denotes the coefficient associated with Interaction  $x_1 x_2$ ,  $\beta_1 x_1$  and  $\beta_2 x_2$  resemble the effects of factors  $x_1$  and  $x_2$ , respectively, and  $\beta_0$  denotes the intercept of the regression model. Regression analysis was carried out based on (1). ANOVA was utilized to calculate the sum of squares (SS), mean squares (MS), F-value, p-value, coefficient of determination ( $R^2$ ), and correlation coefficient ( $|R|$ ). The optimal level of oil temperature, stirring rate, and mixing duration that will maximize the color reduction of RBUVOs was determined using a response surface plot.

## 3. RESULTS AND DISCUSSION

### 3.1. Color reduction test results

Three factorials namely oil temperature, mixing speed and mixing time were analyzed using two-level factorial design. Single response recorded was a color reduction of RBUVOs. The results of the color reduction for all thirteen test runs are demonstrated in Table 2.

### 3.2. Result of the half-normal plot

The half-normal plot and effect list from the 23-factorial design of experiments are shown in Figure 1 and Table 3, respectively. From Figure 1, it can be shown that factors A (oil temperature), B

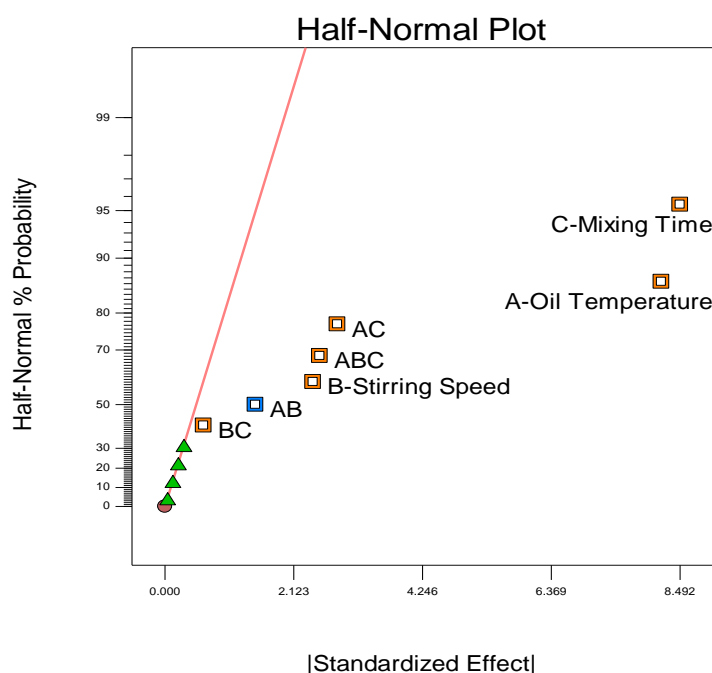
(mixing speed), and C (mixing time). Figure 1 illustrates that factors C (mixing time) and A (oil temperature) are separated from the straight line by a large distance. Similar to factors A and C, interaction AC, interaction ABC, and factor B are similarly situated at a distance from the straight line; however, this distance is not as pronounced. This shows that the model terms factors A, B, C, interaction AC, and interaction ABC are all important. Table 3 contains the effect list, which displays the SS and the percentage contribution for all model terms. The findings show that factor C, with a percentage contribution of 35.00% and a total SS of 144.2240, is the crucial factor. Contrarily, component B only contributes 2.89%, making it very evident that it is the least influential of all the variables. Factor B has an SS of 11.9089. The total squares for component A, interaction AC, and ABC are 133.9614, 16.1787, and 13.0242, accordingly, with contributions of 32.51%, 3.93%, and 3.16%, respectively. Based on the findings, it is clear that component C (mixing time), in comparison to factors B, C, interaction AC, and interaction ABC, considerably contributes more to the color reduction (%). However, it is still considered to be significant that other factors contribute.

Table 2. Mean color reduction for each oil sample

Experimental trial no.	Variable code			Mean color reduction (%)
	A: Oil temperature (°C)	B: Mixing speed (rpm)	C: Mixing time (min)	
1	80 (+1)	1,000 (+1)	30 (-1)	64.74
2	70 (0)	750 (0)	45 (0)	72.00
3	70 (0)	750 (0)	45 (0)	71.56
4	70 (0)	750 (0)	45 (0)	73.29
5	60 (-1)	500 (-1)	30 (-1)	57.60
6	60 (-1)	500 (-1)	60 (+1)	65.16
7	60 (-1)	1,000 (+1)	60 (+1)	67.18
8	70 (0)	750 (0)	45 (0)	72.09
9	80 (+)	500 (-1)	60 (+1)	75.13
10	60 (0)	1000 (+)	30 (-1)	63.45
11	70 (0)	750 (0)	45 (0)	74.19
12	80 (+1)	500 (-1)	30 (-1)	66.98
13	80 (+1)	1,000 (+1)	60 (+1)	79.27

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Color Reduction

- ▲ Error estimates
- A: Oil Temperature
- B: Stirring Speed
- C: Mixing Time
- Positive Effects
- Negative Effects

Figure 1. Half-normal plot produced from the  $2^3$ -factorial design

### 3.3. Result of the ANOVA analysis

Response surface model is used in the analysis of variance (ANOVA) to find the ideal set of mixing process parameters that will produce the RBUVOs with the lowest acidity number. As a result, RBUVOs has their color reduced (%) as a function of oil temperature (variable:  $x_1$ ; unit: °C), mixing speed (variable:  $x_2$ ;

unit: rpm), and mixing time (variable:  $x_3$ ; unit: min). According to a regression model established in accordance with (1), as (3).

$$y = 67.44 + 4.09x_1 + 1.22x_2 + 4.25x_3 - 0.7454x_1x_2 + 1.42x_1x_3 + 0.3184x_2x_3 + 1.28x_1x_2x_3 \quad (3)$$

The regression equation yields a mean and standard deviation of the color reduction of 69.44 and 1.08, respectively. For the regression model terms obtained using ANOVA, Table 4 displays the SS, degrees of freedom (df), MS, F-value, p-value, coefficient of  $R^2$ , and correlation coefficient ( $|R|$ ). A p-value of less than or equal to 0.05 is interpreted as a sign that the model (or model term) is statistically significant by [24], [25]. Since the p-value for the regression model overall is 0.0016, the results demonstrate its significance. Since the p-value is less than 0.05, factor A (oil temperature), factor B (mixing speed), factor C (mixing time), interaction AC, and interaction ABC are significant model terms (0.0004, 0.0332, 0.0004, 0.0205, and 0.0289, respectively). While this model term is not significant, the p-values for the interactions AB (oil temperature and mixing speed) and BC (mixing speed and mixing duration) are 0.1230 and 0.4518, respectively (which is greater than 0.05).

The ANOVA results express that the interactions between the variables AB (oil temperature and mixing speed) and BC (mixing speed and mixing time) do not significantly affect the color reduction in the RBUVOs. In order to significantly increase the color reduction of the RBUVOs, interaction AB (oil temperature and mixing speed) and BC (mixing speed and mixing time) must be combined with all factors. The  $R^2$  value of 0.9858, which shows that the model explains 98.58% variance of the color reduction owing to the fluctuation of the independent variables, further supports the conclusion that the regression model constructed in this study is sufficient, such as oil temperature, mixing speed and mixing time. Additionally, the overall regression model's p-value is 0.0016 (less than 0.05), showing that it is an insignificant model. The actual and anticipated color reduction are correlated, as indicated by the  $|R|$ . The general rule is that a substantial correlation exists between the observed and projected values if  $|R|$  is close to 1. The regression model's  $|R|$  value is determined to be 0.9858, demonstrating the strong correlation between the anticipated acidity value and the results of the experiments.

Figure 2(a) illustrates that for RBUVOs, the largest color reduction (79.27%) occurs when the oil is heated to 80 °C, the stirring is done at 1,000 rpm, and the mixing time is 60 min. Accordingly, these are the maximum settings for the mixing process parameters since the highest oil temperature, mixing speed, and mixing time results in the highest color reduction levels. It is interesting to note that the lowest oil temperature, mixing speed, and mixing time (60 °C, 500 rpm, and 30 minutes, respectively) all result in the lowest color reduction (57.60%), as shown in Figure 2(b). This suggests that compared to mixing speed, the oil temperature and mixing time possess a more significant impact on the color reduction of RBUVOs. The results demonstrated that the maximum level of mixing process parameters had significantly improved the lightness of RBUVOs and significantly reduced color pigment in RBUVOs.

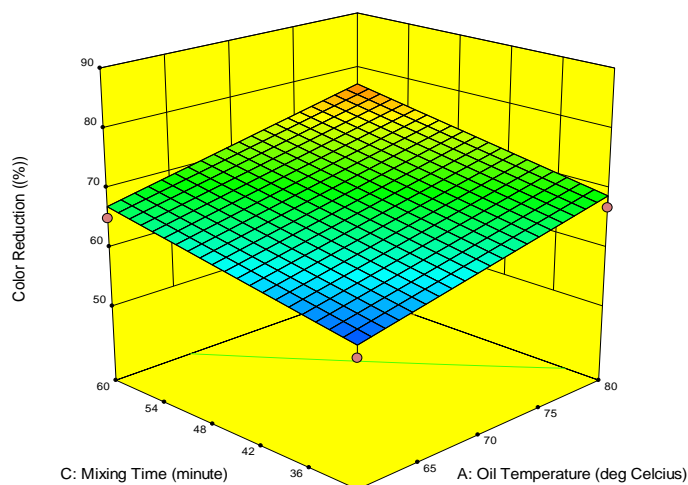
Table 3. Effect list employed from the 2<sup>3</sup>-factorial design of experiments

Model term	Standardized effects	Sum of squares	Percentage contribution (%)
A	8.1842	133.9614	32.51
B	2.4402	11.9089	2.89
C	8.4919	144.2240	35.00
AB	-1.4908	4.4451	1.08
AC	2.8442	16.1787	3.93
BC	0.6368	0.81097	0.20
ABC	2.5519	13.0242	3.16

Table 4. A regression model with factorial response surface fitting ANOVA results

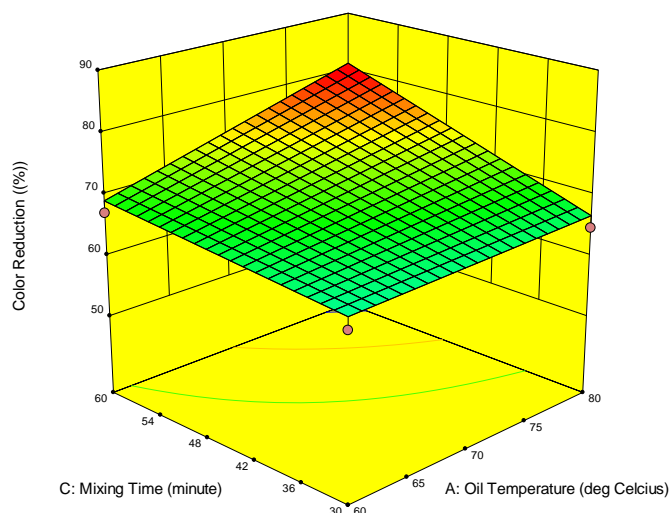
Source	SS	df	MS	F-value	p-value	$R^2$
Overall model	324.55	5	63.8594	38.5764	0.0016	0.9858
A-oil temperature	133.9614	1	133.9614	80.9238	0.0004	
B-mixing speed	11.9088	1	11.9089	7.1940	0.0332	
C-mixing time	144.22	1	144.2240	87.1233	0.0004	
AB	4.45	1	4.45	3.80	0.1230	
AC	16.18	1	16.1787	9.7733	0.0205	
BC	0.8110	1	0.8110	0.6937	0.4518	
ABC	13.024	1	13.0242	7.8677	0.0310	
Pure error	4.68	4	1.17	-	-	
Total correlation	412.02	12				

Design-Expert® Software  
 Factor Coding: Actual  
 Color Reduction ((%))  
 ● Design points below predicted value  
 79.2695  
 57.6014  
 X1 = A: Oil Temperature  
 X2 = C: Mixing Time  
 Actual Factor  
 B: Stirring Speed = 500



(a)

Design-Expert® Software  
 Factor Coding: Actual  
 Color Reduction ((%))  
 ● Design points below predicted value  
 79.2695  
 57.6014  
 X1 = A: Oil Temperature  
 X2 = C: Mixing Time  
 Actual Factor  
 B: Stirring Speed = 1000



(b)

Figure 2. 3D response surface plot; to identify (a) maximum and (b) minimum percentage of color reduction from mixing process parameters

#### 4. CONCLUSION

It has been illustrated in this study that the two-level factorial design of experiments is a useful technique for determining the highest level of oil temperature, mixing speed, and mixing time mixing process parameters that will maximize the color reduction of RBUVOs. The primary benefit of the two-level  $2^k$  factorial design of experiments approach is the ability to identify from fewer test runs the factors that will significantly affect the color reduction of RBUVOs, as shown by the percentage contribution of each factor. This greatly cuts down on time and expense, which are normally drawbacks to traditional experimental procedures. The results from the  $2^3$  factorial designs show that mixing time and oil temperature, with percentage contributions of 35.00% and 32.51%, respectively, have the most significant effects on the color reduction of RBUVOs. According to the response surface plot created, the greatest level of mixing process parameters for oil temperature, mixing speed, and mixing speed that will result in the highest color reduction are 80 °C, 1,000 rpm, and 60 min. It was discovered that the model is sufficient to predict the highest color reduction of RBUVOs as a function of the oil's temperature, the mixing speed, and the mixing time setting.

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